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**OFF-AXIS CREEP BEHAVIOR OF
OXIDE/OXIDE NEXTEL™720/AS-0
(PREPRINT)**

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Off-axis Creep Behavior of Oxide/Oxide Nextel™720/AS-0

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Abstract

Oxide/Oxide Ceramic Matrix Composites (CMC) are currently being demonstrated in high-temperature aerospace applications where their oxidation resistance is critical to a successful design. Many applications are engine components that are axisymmetric in shape and subject to axisymmetric thermal and mechanical loadings. Traditionally woven CMC materials used in these components are typically made from 0°/90° fiber architectures. In many cases the highest stresses are not always coincident with the orientation of the reinforcing fibers. Therefore, these components may experience stress states that approach the off-axis tensile and creep strengths of the material. The oxide/oxide CMC investigated in this study was Nextel™720/AS. Nextel™720/AS is composed of an Alumina-Silica matrix reinforced with an eight-harness satin weave (8HSW) of Nextel™720 fibers. Tensile and creeps tests at 1100°C on ±45° Nextel™720/AS were performed to characterize the off-axis material behavior. The ±45° orientation has approximately two thirds the ultimate tensile strength of the 0°/90° orientation. However, the tensile toughness of the ±45° orientation was higher than the 0°/90° orientation by a factor of 2X. The creep rupture strength at 100 hrs for the ±45° orientation was approximately one third that of the 0°/90° orientation.

Keywords: A. Ceramic matrix composites, ±45° orientation; B. Creep; C. Creep rate

Introduction

Damage tolerant ceramic matrix composites (CMC) that exhibit relatively little degradation in mechanical properties at elevated temperature are being evaluated for high temperature aerospace applications. Advances in CMC processing have enabled lower-cost fabrication techniques that provide the capability to produce complex shaped components. Many of these complex shapes have fillets, corners, holes and other structural features that develop multiaxial stress states under thermal and mechanical loading.

Typically, characterization of new composite materials tends to focus on properties aligned with the direction of the reinforcing fiber such as strength, moduli and ductility. This may satisfy a particular design or material requirement but it is not sufficient, in general, for a structural material. Although a good understanding of the stresses and temperatures in the intended application may be known it is often difficult to precisely know the extremes and the perturbations that a particular component or feature may experience. Therefore it is desirable to have a structural material that is damage tolerance, stable at elevated temperatures and quasi-isotropic with respect to pertinent material properties.

Often engine applications require that material properties be maintained at elevated temperature and under combustion environment conditions for long durations.

Nextel™720/AS CMC has an inherent resistance to oxidation and therefore exhibits little or no degradation in mechanical properties for long term exposures below 1100°C. Several authors [1-7] have reported that oxide/oxide CMC exhibit excellent tensile, fatigue and creep properties at room and elevated temperatures. Monotonic tensile behavior at room and elevated temperature (1100°C) on $\pm 45^\circ$ Nextel™720/AS [10,11] have shown that the ultimate strength and strain to failure are a function of test temperature. This is in contrast to the $0^\circ/90^\circ$ tensile behavior. This investigation focuses on the sustained load (creep) behavior of the $\pm 45^\circ$ orientation at 1100°C. This paper characterizes the differences in the material behavior of the Nextel™720/AS in the $0^\circ/90^\circ$ and $\pm 45^\circ$ orientations.

Material

The Nextel™720/AS oxide/oxide CMC was manufactured by ATK COI Ceramics, Inc, of San Diego, CA. The Nextel™720 fibers, which are produced by the 3M Company of St. Paul, MN [15], are approximately 12 μm in diameter and have a nominal composition of 85% Al_2O_3 and 15% SiO_2 . The addition of Mullite ($3 \text{ Al}_2\text{O}_3 \cdot 2 \text{ SiO}_2$) to the Nextel™720 fiber provides improved creep resistance over the Nextel™610 fiber that is a single-phase composition of $\alpha\text{-Al}_2\text{O}_3$. The Nextel™720 fibers have an elastic modulus of 260 GPa and a tensile strength of 2100 MPa for a 25 mm gage length [8]. Approximately 400 fibers were bundled together into a tow. Multiple tows are woven together into a balanced eight-harness-satin weave (8HSW) cloth.

The porous alumina-silica (AS) matrix forms a weak bond to the fibers without an engineered interphase. The weak fiber-matrix bond combined with the porous matrix produces a material structure that cannot develop sufficient strain energy to propagate a single dominate self-similar crack. Relative movement between the fiber and matrix enables crack deflection and increased toughness resulting in a CMC with damage tolerance behavior. The porous matrix contains approximately 87 wt% single crystal submicron alumina suspended in 13 wt% silica. The nested plies were warp aligned during lay-up with alternating plies rotated about the warp direction so that fill fibers were matched with fill fibers and warp fibers matched with warp fibers. This lay-up technique matches fabric texture on neighboring plies and minimizes panel warpage. The 2D Nextel™720 fabric was prepregged with alumina powder and a silica forming polymer before stacking. The laminate was then warm molded to produce the green state ceramic tile. Sintering the green tile in air at 1000°C removed the organic binders and produced a porous alumina-silica matrix. Section samples of panels used in this study show that the fiber volume fraction range was 42 - 50%. Figure 1 show microcracks distributed throughout the matrix rich regions of the composite as a result of panel shrinkage that occurred during processing. The panels used for this study were 300 mm x 300 mm and composed of 12 plies.

Experimental Procedure

The dogbone specimen geometry, shown in Fig. 2, was used for unnotched tensile and creep tests in this study. The nominal dimensions of the specimens were, thickness = 2.5 mm, length = 150 mm and grip width = 16 mm. The gage section width of 10 mm provides a 37.5% reduction in area to promote gage section failure. The gage section width is also greater than the repeating cell size of the 8HSW cloth, which is approximately 9 mm. The 18 mm uniform width gage length permits placement of a

conventional elevated-temperature extensometer and provides a volume averaging of fiber and matrix properties that will be used for development of a macroscopic material model. All specimen dimensions were measured prior to testing.

The specimen ends were tabbed with fiberglass to provide a flat uniform surface for the grip surfaces and to minimize the possibility of grip failure. The specimens were mounted in a precisely aligned, rigid grip system that minimized specimen bending and rotation [16]. A clamshell furnace with silicon-carbide heating elements and four-zone temperature control were used for all elevated temperature tests. Four S-type thermocouples were used to measure and control the temperature in the gage section of the test specimen. Two thermocouples were on the top surface and two were on the bottom surface. Four-zone temperature control provided the capability of reducing the temperature gradients through the specimen thickness and along the loading axis of the specimen. Thermal profile maps on the specimen show that the specimen was uniformly heated, $\pm 5^\circ\text{C}$, over the entire gage section of the specimen.

All tensile tests were performed in displacement control at a rate of 0.05 mm/s generated by a 16-bit D/A function generator. The tensile test duration was typically less than 10 seconds and therefore time dependent creep behavior did not contribute to the measured deformation. Creep tests were performed in load control at a loading rate of 20 MPa/s up to the hold stress for all creep tests. Extensometry measurements were made on both sides of the test specimen to verify that specimen bending and hence stress gradients induced in the gage section were negligible. Also, prior to starting any test, room temperature and elevated temperature modulus measurements were made by loading the sample in stress control to approximately 20 MPa, well below the proportional limit. Tensile and creep tests were started if and only if the room temperature and elevated temperature modulus values agreed with expected results and the modulus values from the two opposing extensometers were in agreement with each other, hence no bending in gage section. Applied load, temperature and extensometry data were collected during all tensile and creep tests. All tests were conducted in laboratory air using a computer controlled servo-hydraulic horizontal test system [16, 17].

Experimental Results – Monotonic Tensile Behavior

Monotonic tensile tests at room and elevated temperatures on specimens with woven fiber aligned in $0^\circ/90^\circ$ orientation are shown in Fig. 3. At temperatures up to and including 1100°C there was no degradation in the elastic modulus or ultimate strength of the $0^\circ/90^\circ$ orientation. These modulus values ($E \sim 62\text{GPa}$) are consistent with data reported on similar oxide/oxide systems [1, 11, 14, 22] at room and elevated temperature. The 1200°C tensile data show that ultimate strength was preserved while initial elastic modulus slightly decreased and total deformation to failure increased. Tensile strength of NextelTM720 fibers measured by Wilson et al. [8] show approximately a 20% decrease in strength at 1200°C compared to 1100°C and below. The decrease in fiber strength and retained composite tensile strength at 1200°C suggest the presence of residual stresses at the lower temperatures. This is also supported by the decrease in elastic modulus of the composite which is unaffected by the presence of the residual stress. Zawada et al. [18] reported tensile residual stresses in the Alumina-Silica matrix on the order of 200 MPa for a similar oxide/oxide CMC at room temperature.

Monotonic tensile tests were also conducted on dogbone specimens with woven fibers mats aligned in $\pm 45^\circ$ orientations. The axial stress versus axial strain response for the $\pm 45^\circ$ orientation at room and elevated temperatures are shown in Fig. 4. Tensile data

reported by Antti & Lara-Curzio [10] on the same oxide/oxide CMC are shown for room temperature and 1000°C. In contrast to the 0°/90° data from Fig. 3, the ±45° orientation exhibited a strong temperature dependent deformation response. Although the initial elastic modulus remained the same for this range of temperatures, the ultimate tensile strength and strain to failure increased with increasing temperature.

The increase in ultimate tensile strength with temperature provides additional supporting evidence for the existence of residual stresses in the CMC at room temperature. Since the fibers in the ±45° orientation are not continuous from one end of the specimen to the other, composite deformation behavior was not driven by the strength or ductility of the Nextel720 fibers.

The axial stress versus axial strain for the 0°/90° and ±45° orientations at 1100°C are shown together in Fig. 5. As expected, the 0°/90° orientation, which has continuously reinforced fibers between the grips has a higher ultimate tensile strength and lower strain to failure than the ±45° orientation. The mechanical behavior of the CMC is driven primarily by the high strength, high modulus and low ductility fibers. However, the initial elastic modulus, measured up to 50 MPa, show that the two orientations have similar elastic moduli, approximately 62 GPa. This is in contrast to the modulus results reported by Levi et al. [6] on Nextel™610 oxide/oxide CMC where the elastic modulus was strongly orientation dependent. The different off-axis behavior between the two CMC systems is that the Nextel™610 fiber has a much higher modulus than the Nextel™720 fiber, 380 and 260 GPa, respectively. Zuiker [20] and Tandon et al. [19] have predicted oxide matrix modulus values of approximately 60 GPa and less. The higher modulus Nextel™610 fiber will produce a more pronounced orientation dependent elastic modulus in the CMC. For stresses are above ≈ 50 MPa, the ±45° orientation exhibits a large decrease in stiffness while the 0°/90° orientation maintains initial stiffness to failure.

The fracture surfaces for the 0°/90° and ±45° tensile specimens at 1100°C are shown in Figs. 6a and 6b, respectively. The fracture surface profiles for the two orientations are coincident with the fiber weave orientations. For the 0°/90° orientation the fracture plane is normal to the applied load which is typical for a brittle material. In contrast, the ±45° orientation fracture plane was at a 45° angle with respect to the applied load which is typical for a softer or more ductile material when the ultimate shear stress is reached. Since the reinforcing fibers in the reduced gage section of the ±45° sample are not attached to either gripped end of the specimen it is relatively easy for the fiber tows to slip past one another at a 45° angle and fail the matrix. The area under the entire engineering stress-strain curve of Fig. 5, the tensile toughness, is a measure of the absorbed energy or damage accumulation in the material. For the two stress-strain curves in Fig. 5 the tensile toughness of the 0°/90° and ±45° orientations were 0.30 and 0.71 MJ/m³, respectively. Although the ±45° orientation had a lower ultimate strength than the 0°/90° orientation it required more than 2X inelastic work to fail the specimen. This was primarily due to the fact that the damage occurred over a larger volume of material.

Experimental Results – Sustained Load (Creep) Behavior

Based on the monotonic tensile results of the previous sections shown in Figs. 3-6, and the author's previous work [1, 12, 13] on oxide/oxide CMC, the sustained load (creep) behavior of the material in the ±45° orientation was investigated. The total strain versus time of the ±45° Nextel720/AS material subjected to sustained load (creep) at 1100°C are shown for four stress levels (50, 60, 70, 80 MPa) in Fig. 7. The deformation behavior shows a strong influence of applied stress on the time to rupture. Also, a

decrease in total strain occurs with an increase in applied stress. These results are for a stress range that corresponds to 40 - 65% of the $\pm 45^\circ$ ultimate tensile strength. Previous creep studies [1] on the $0^\circ/90^\circ$ orientation exhibited very little creep deformation at 1100°C . Only when the applied stress was at greater than 80% of the $0^\circ/90^\circ$ ultimate tensile strength was there any measurable time dependent deformation. Clearly time dependent creep is activated at lower stresses and presumably lower temperatures in the $\pm 45^\circ$ orientation compared to the $0^\circ/90^\circ$ orientation.

The NextelTM720 fibers exhibit stable deformation behavior up to approximately 1100°C for extended periods of time [8]. Carelli et al. [21] have demonstrated that the usage temperature of this CMC is ultimately limited by the alumina-silica matrix properties. Long-term exposure at 1100°C sinters the matrix and causes the matrix to clamp to the fibers. This negates the intentional weak interface that was designed for damage tolerance by deflecting matrix cracks. Hence, the maximum usage temperature for long-term exposure of this composite material is approximately 1050°C which is in agreement with aging studies by Jurf and Butner [23].

The stress dependence of the $\pm 45^\circ$ orientation creep deformation shown in Fig. 7 along with the thermal aging behavior of the alumina-silica matrix [23] indicated a probably temperature dependence as well. Consequently, the temperature dependent creep behavior was investigated by conducting sustained load (creep) tests at constant stress with step increases in temperature. A minimum starting temperature of 900°C was chosen with temperature steps of 50°C taken up to a maximum of 1100°C . The temperature was held constant until it appeared that the material had achieved a stable constant creep rate. After achieving a constant creep rate the temperature was rapidly increased to the next higher temperature and the process repeated until failure or the test temperature of 1100°C was reached. Figure 8 shows the total strain versus time for a step temperature creep test that was conducted at an applied stress of 70 MPa. The total strain to failure from the step temperature test at 70MPa was similar to the 70MPa isothermal test in Fig.7. The time to rupture was clearly affected by the prior lower temperature strain history. The extended time at lower temperature contributed to the overall increase in rupture time and approximately 40% of the total deformation. As expected, the slopes of the strain versus time curves, i.e., the creep rate, increased with each increase in temperature.

The 900 and 950°C portions have a relatively flat deformation response whereas the 1050 and 1100°C portions show strain rates comparable to the creep rates at 60 and 70MPa at 1100°C of Fig. 6. A similar stepped temperature test was conducted at 90MPa with the results shown in Fig. 9. As expected the time to failure for the 90MPa test was shorter than the 70MPa test for similar time and temperature exposures. Also consistent with the higher stress, shorter life test was a decrease in total strain to failure, approximately 40% compared to the 70MPa stepped temperature test. At every temperature the steady state strain rate was higher for the 90MPa test than the 70MPa test.

Strain rate dependence on stress is often described by a power law model. The strain rate dependence on temperature is best modeled using the Arrhenius rate equation. Combining the power law equation and Arrhenius equation results in the creep rate equation shown below where (A , n , ΔH_A) are fitting parameters, (R) is the universal gas constant, (k) is Boltzmann's constant, (T) is temperature in Kelvin and (σ) is stress in MPa.

$$\dot{\epsilon}_c = \frac{A}{kT} \sigma^n \exp\left(\frac{-\Delta H_A}{RT}\right) \quad (1)$$

The strain rate coefficient (A), the stress exponent (n), and the activation energy (ΔH_A) were determined from the change in stable strain rates for step increases in temperature as shown by the equation above. Parameters A , n and ΔH_A were determined from a least squares fit to the experimentally measured strain rate data.

Figure 10 shows the minimum creep rate versus the inverse of the test temperature, in Kelvin, for all the $\pm 45^\circ$ orientation step temperature creep data. The symbols represent the measured creep rates and the lines are the calculated creep rates based on the power law model. The near linearity of the data suggests that deformation is dominated by a single mechanism for this range of temperatures and stresses.

A comparison of minimum creep rate versus applied stress for the $0^\circ/90^\circ$ and $\pm 45^\circ$ orientations is shown in Fig. 11. Creep results from constant temperature tests, shown as symbols, from Zawada et al. [1] and Lara-Curzio and More [14] on $0^\circ/90^\circ$ NextelTM720/AS-0 at 1100°C and 1200°C , respectively, show a much lower creep rate compared to the $\pm 45^\circ$ data at 1100°C from this study (Fig. 7). The solid red line represents the prediction of the minimum creep rate for the $\pm 45^\circ$ orientation using Eq. 1 which was fit to the step temperature creep results shown in Figs. 8 & 9. For a creep stress, $\sigma \approx 90\text{MPa}$, the $0^\circ/90^\circ$ strain rate is over 1000X slower than $\pm 45^\circ$ at 1100°C . Even at 1200°C , which is above the usage temperature for the NextelTM720/AS-0, the $0^\circ/90^\circ$ strain rate is an order of magnitude lower than the $\pm 45^\circ$ orientation at 1100°C . The minimum creep rate is clearly dependent on the orientation of the reinforcing fibers.

Figure 12 shows the applied net section stress versus creep rupture time for unnotched and double edge notch $0^\circ/90^\circ$ and unnotched $\pm 45^\circ$ Nextel720/AS-0 at 1100°C . Previous studies [13,14] on the $0^\circ/90^\circ$ orientation have shown the double notched geometry exhibits a reduction in creep rupture life when compared to the unnotched geometry. The unnotched $\pm 45^\circ$ orientation data in Fig. 12 show a further reduction in creep rupture life from the notched $0^\circ/90^\circ$ data. To achieve a rupture life of 100 hours at 1100°C the net section stress for the unnotched $0^\circ/90^\circ$, notched $0^\circ/90^\circ$ and unnotched $\pm 45^\circ$ should be less than 155, 95 and 55 MPa, respectively. Clearly, this knockdown in the rupture life of the $\pm 45^\circ$ compared to the $0^\circ/90^\circ$ orientation at 1100°C limits the applicability of this material for components subjected to multiaxial states of stress.

It is also clear that a Cartesian fiber architecture such as a $0^\circ/90^\circ$ is not well suited for construction of axisymmetric components where the principal stress axes are not coincident with the orientation of the reinforcing fiber weave. The fracture surfaces for the $0^\circ/90^\circ$ and $\pm 45^\circ$ creep tests at 1100°C with similar rupture lives, approximately 20 hours, are shown in Figs. 13a and 13b, respectively. The stress levels were 160 and 70 MPa, for the $0^\circ/90^\circ$ and $\pm 45^\circ$ orientations, respectively. The fracture surface profiles for both orientations are normal to the applied stress. For the $0^\circ/90^\circ$ orientation the fracture plane for the monotonic tensile test and the creep test were the same. In contrast, the $\pm 45^\circ$ orientation fracture plane was at a 45° angle for the monotonic tensile test while the creep test fracture plane was normal to the applied stress. It has been shown that long term thermal exposure sinters the matrix and the fiber and matrix becomes clamped to the fiber. It appears that the clamping of the matrix to the fiber has changed the fracture behavior of the $\pm 45^\circ$ orientation. Therefore long term exposure to elevated temperature reduces the damage tolerance of the $\pm 45^\circ$ orientation.

Summary

The creep rupture behavior of oxide/oxide NextelTM720/AS CMC with 2D fiber mat orientations $0^\circ/90^\circ$ and $\pm 45^\circ$ were investigated. Tensile tests on $0^\circ/90^\circ$ and $\pm 45^\circ$ orientations at 1100°C show similar elastic moduli, however they have different ultimate

strengths and strains to failure. Results of creep tests show that the secondary creep strain rate of the $\pm 45^\circ$ orientation can be as high as 1000X that of the $0^\circ/90^\circ$ orientation at 1100°C . The ratio of the creep rupture strength at 100 hours and 1100°C of $\pm 45^\circ$ to $0^\circ/90^\circ$ is 0.3 for unnotched specimens. Stable creep behavior and possibly the maximum usage temperature of the $\pm 45^\circ$ orientation is about 1050°C

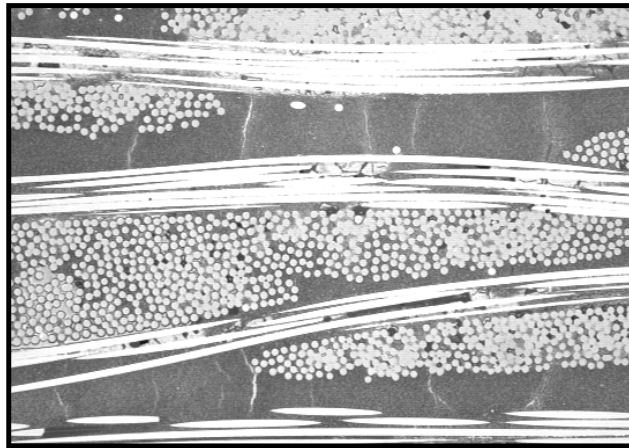
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200 μm

Figure 1. Microstructure of (0°/90°) 8HSW Nextel™720/AS-0 microstructure highlighting the matrix cracks that form during processing.

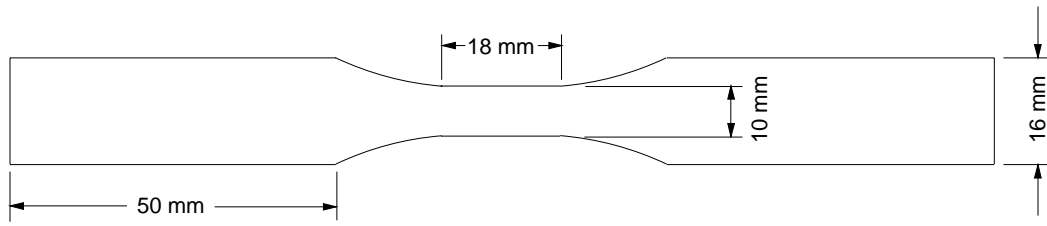


Figure 2. Dogbone specimen test geometry.

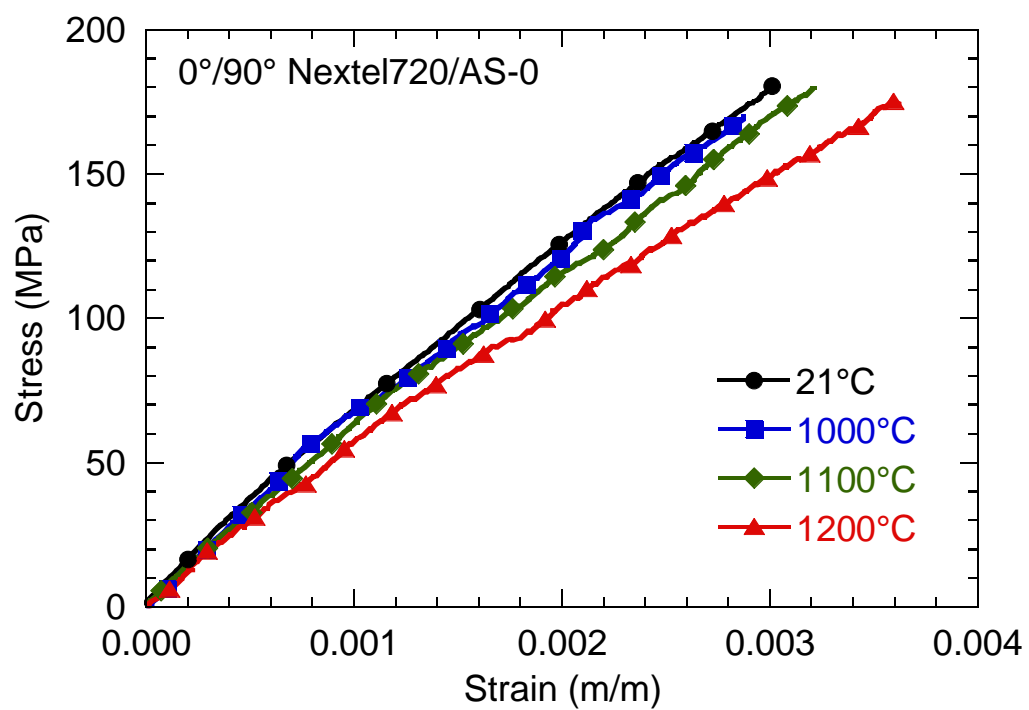


Figure 3. Stress versus strain for NextelTM720/AS-0 oriented in the 0°/90° RT-1200°C.

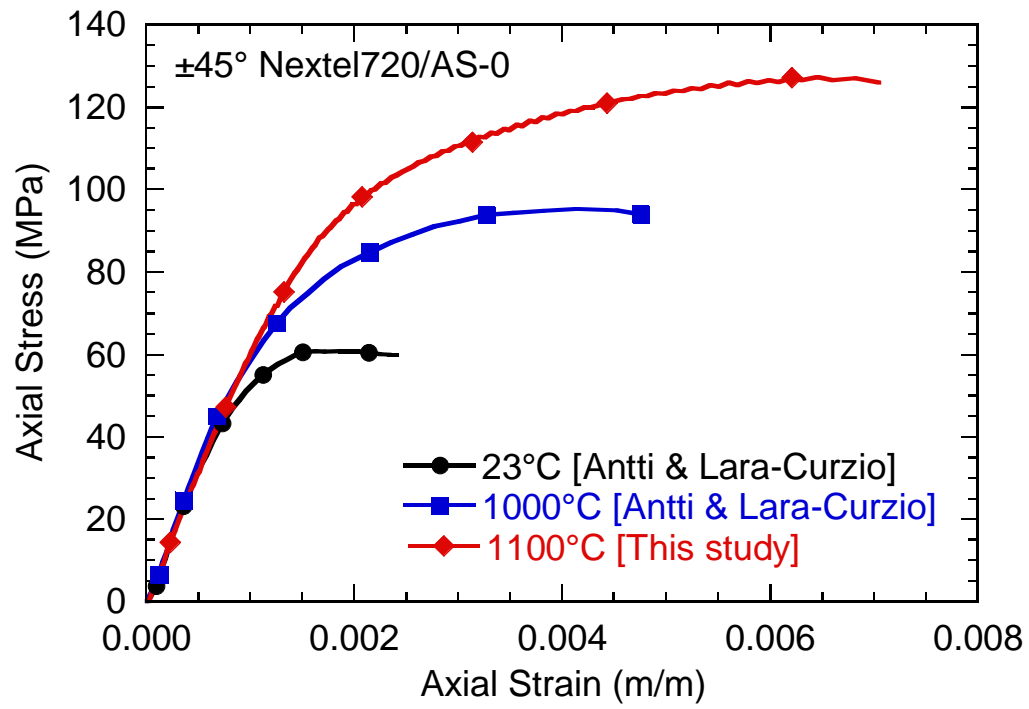


Figure 4. Stress versus strain for NextelTM720/AS-0 oriented in the $\pm 45^\circ$ RT-1100°C.

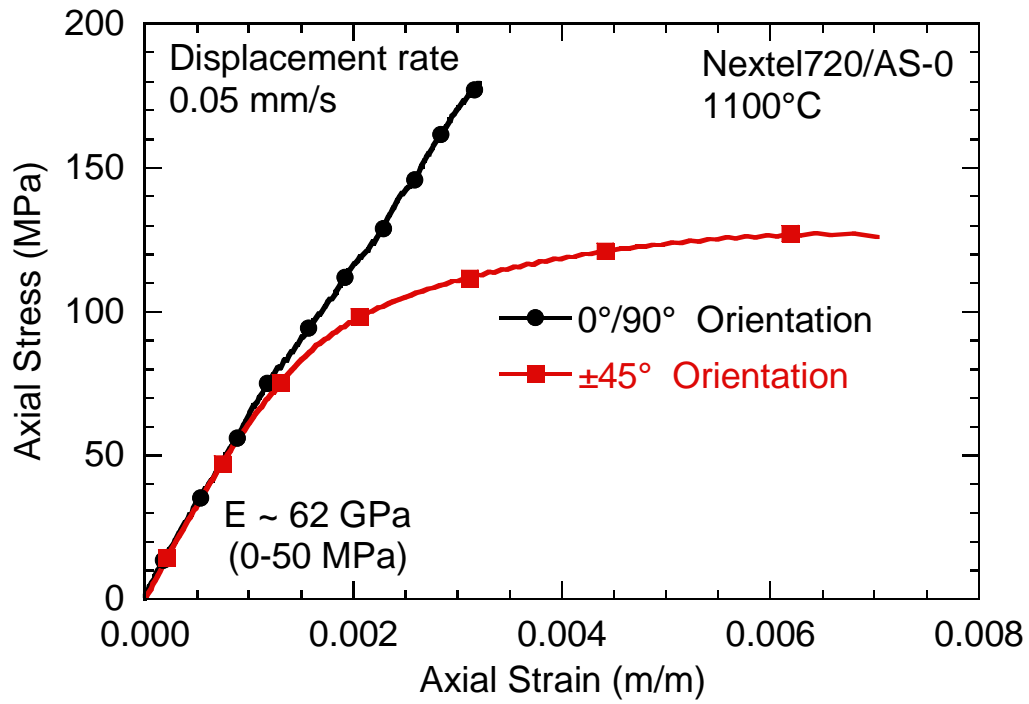


Figure 5. Stress versus strain for NextelTM720/AS-0 oriented in the 0°/90° and ±45° at 1100°C.

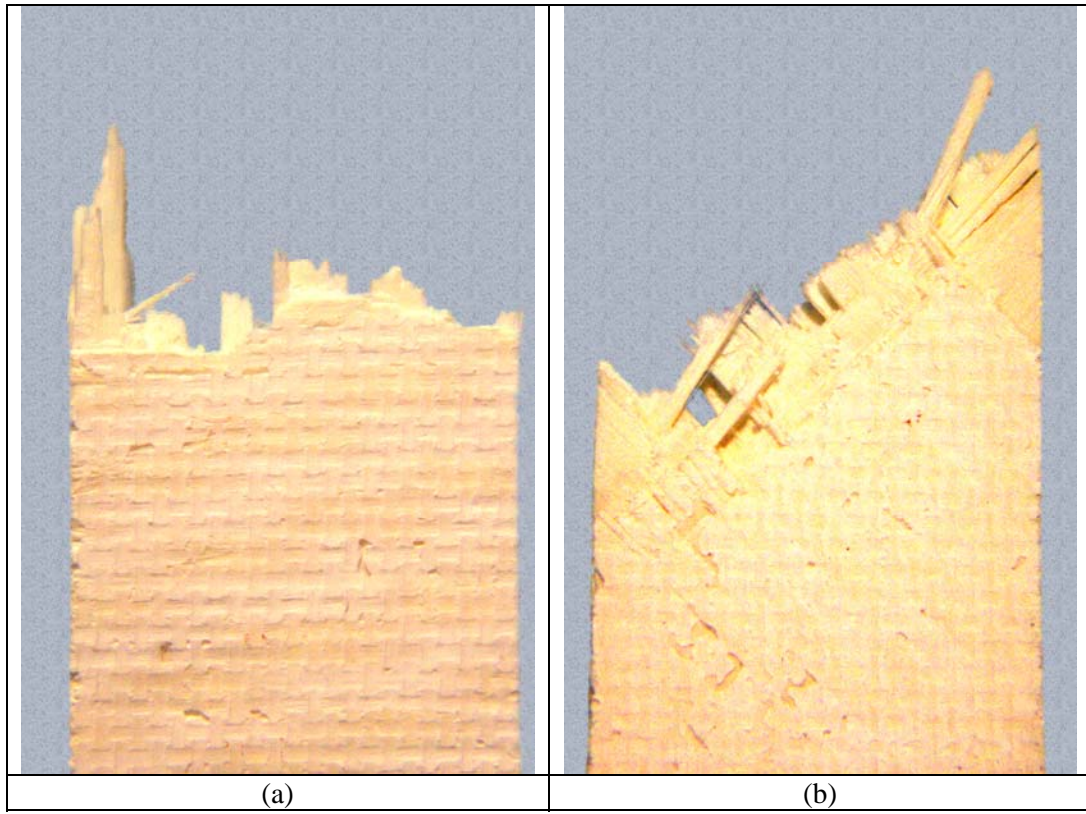


Figure 6. Fracture surface of tensile tested Nextel™720/AS-0 at 1100°C (a) 0°/90°, (b) ±45°.

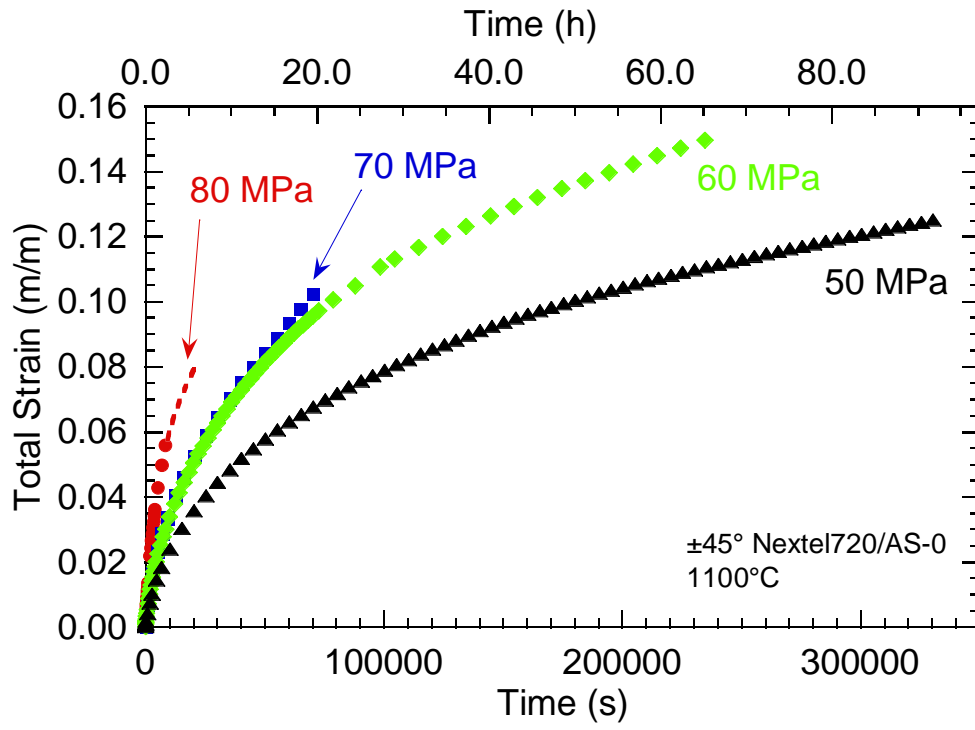


Figure 7. Total strain versus time for $\pm 45^\circ$ Nextel720/AS-0 at 1100°C.

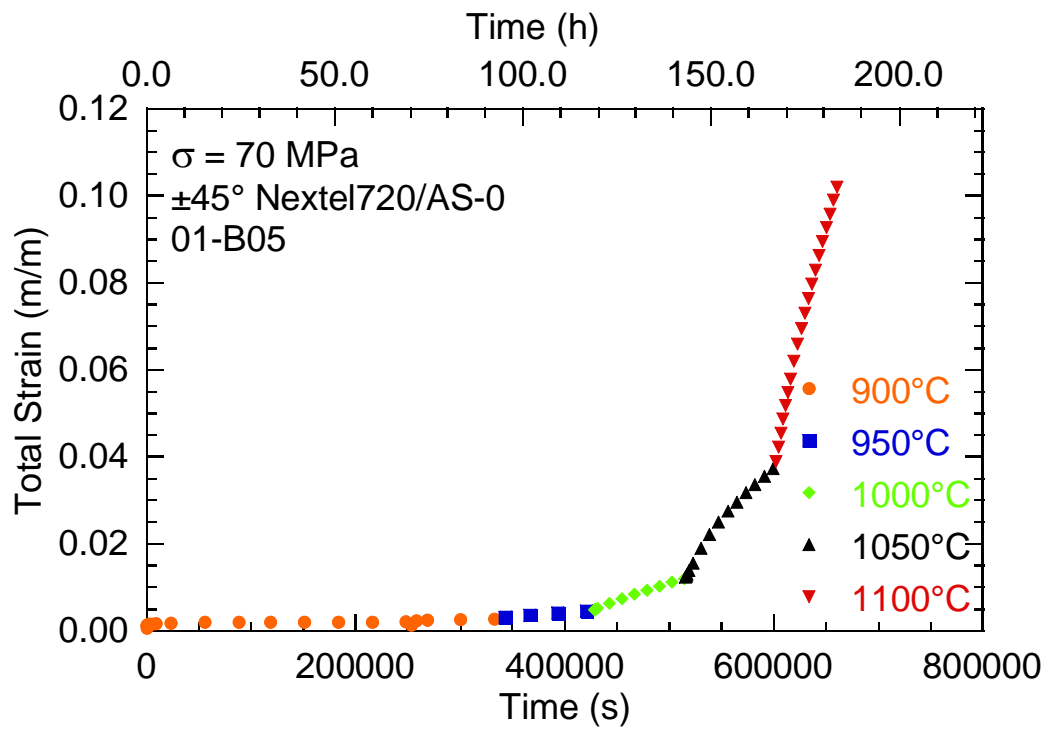


Figure 8. Total strain versus time for $\pm 45^\circ$ Nextel720/AS-0 for stepped temperature (900-1100°C) creep test at 70MPa.

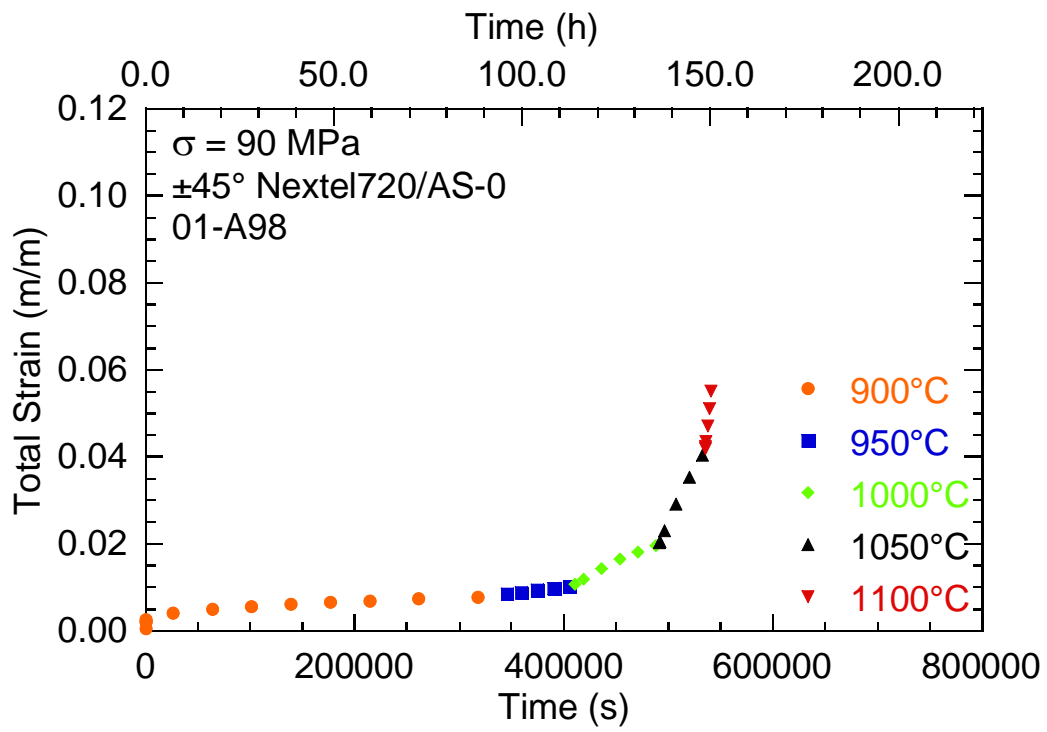


Figure 9. Total strain versus time for $\pm 45^\circ$ Nextel720/AS-0 for stepped temperature (900-1100°C) creep test at 90MPa.

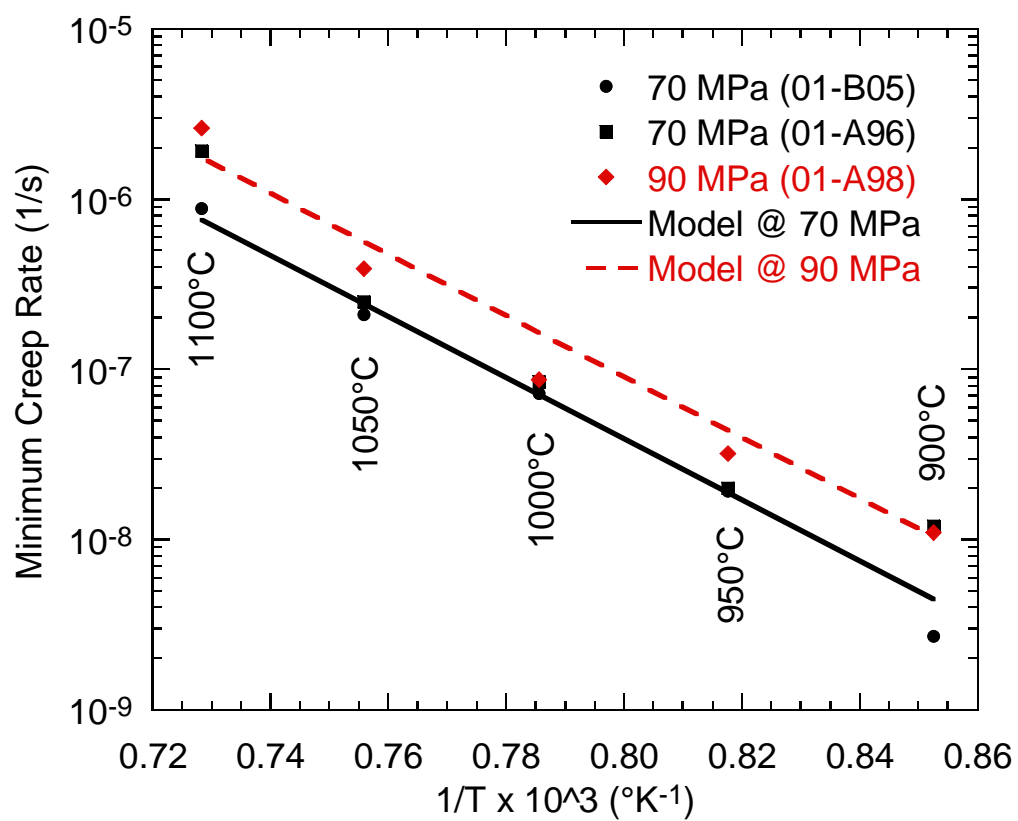


Figure 10. Strain rate versus $1/T$ for creep of $\pm 45^{\circ}$ Nextel720/AS-0.

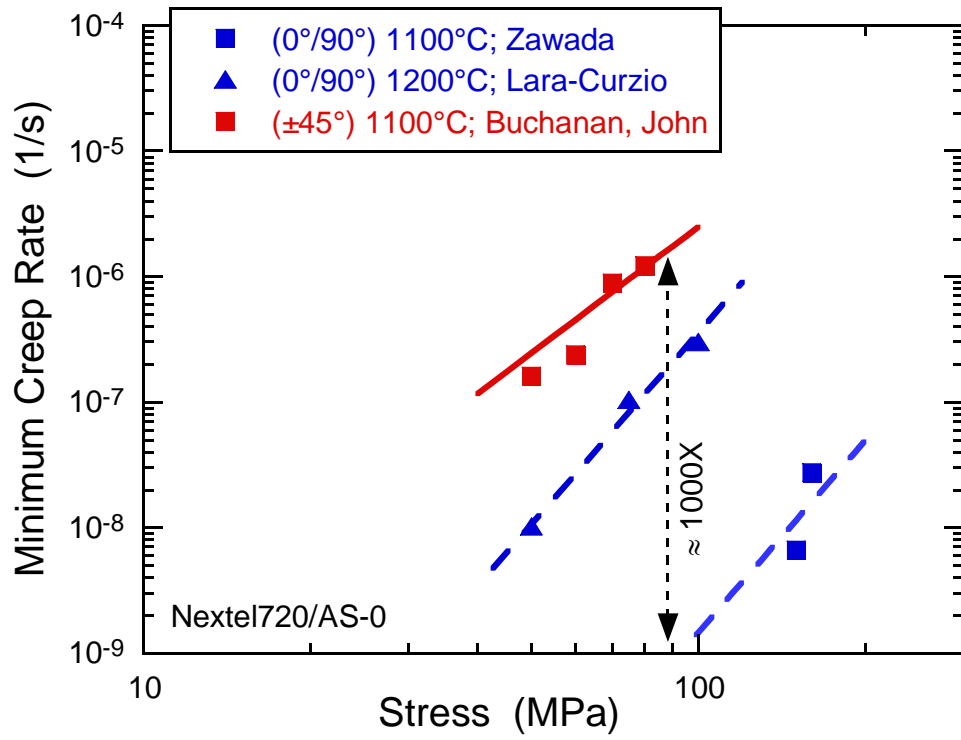


Figure 11. Minimum strain rate versus stress for creep of 0°/90° and ±45° Nextel720/AS-0.

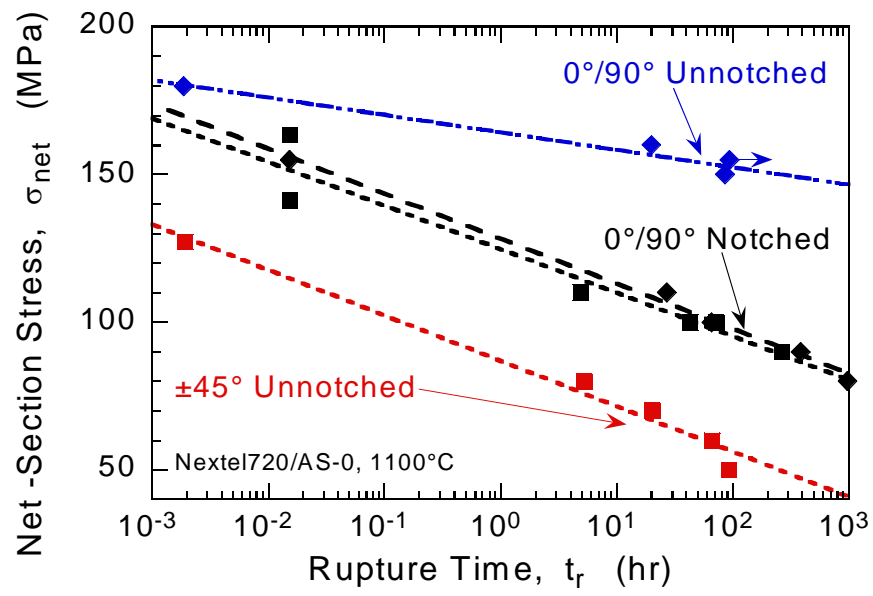


Figure 12. Creep rupture behavior of 0°/90° and ±45° Nextel720/AS-0 at 1100°C.

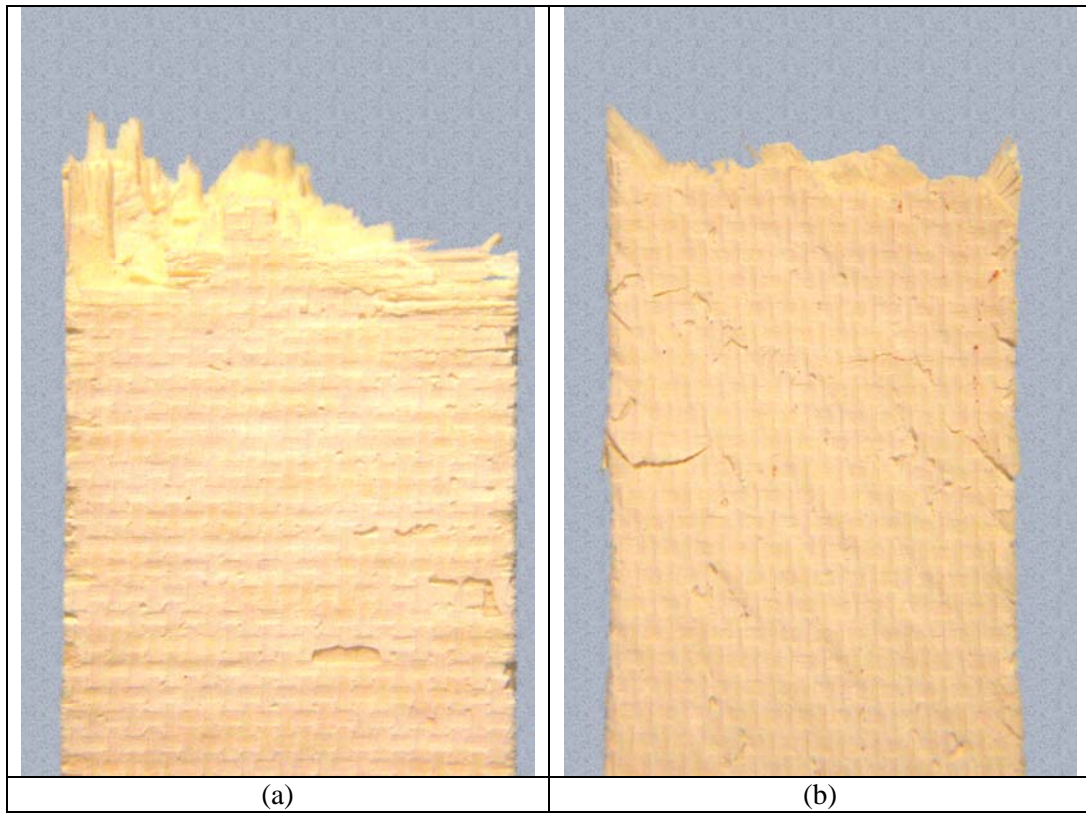


Figure 13. Fracture surface of sustained load (creep) tested Nextel™720/AS-0 at 1100°C (a) 0°/90° , $\sigma = 160$ MPa, $Tr = 20$ hrs, (b) $\pm 45^\circ$, $\sigma = 70$ MPa, $Tr = 20$ hrs.